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IS 4028 (1992): Beeswax, Bleached for Cosmetic Industry
[PCD 19: Cosmetics]



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भारतीय मानक

बीज्वैक्स ब्लीचड सौंदर्य प्रसाधन उद्योग के लिए – विशिष्ट

(तीसरा पुनरीक्षण)

Indian Standard

BEESWAX, BLEACHED FOR COSMETIC
INDUSTRY — SPECIFICATION

(*Third Revision*)

UDC 638.171 : 665.58

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BUREAU OF INDIAN STANDARDS
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NEW DELHI 110002

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FOREWORD

This Indian Standard was originally published in 1965 and first revised in 1977. Subsequently, a second revision was done in 1982. In this third revision, a new requirement of iodine value is being incorporated to check the addition of hydrogenated castor oil to the beeswax and also to improve the keeping quality of the product. However, beeswax with higher iodine value is prone to rancidity at a faster speed. That is why a limiting value of 10 as *Max* has been incorporated.

Secondly for determining presence of ceresin, paraffin and other waxes in beeswax, certain users had expressed difficulty in procuring aldehyde free rectified spirit. A procedure to prepare aldehyde free alcohol in laboratory has been incorporated in this revision. Procedure and interpretation for this test has also been modified. Besides, requirement for relative density has been deleted in this revision as it has been found to be a redundant requirement, not serving any useful purpose. While a requirement for solubility has been included in this revision.

All other changes considered necessary in order to align the standard with the other in the series for cosmetic raw materials have also been incorporated in this revision.

Bleached beeswax is the purified wax obtained from the honeycomb of the bee *Apis dorsata* Linn., fam. Apidae and possibly other species of *Apis* (*A. indica*, *A. Florea*, etc). The material is obtained from honeycombs after removal of honey and subjecting the wax to a preliminary refining, such as melting, scumming decantation and moulding; further purification by melting and finer filtration; and subsequent bleaching, solar or chemical.

Beeswax is used in cosmetic industry in cold creams, lipsticks, brilliantines, depilatories and liquid hair dressings.

The quality of commercial beeswax has been covered in IS 1504 : 1974 Specification for beeswax (*second revision*) which is expected to provide guidance in selecting the raw material of right quality for the manufacture of bleached beeswax for cosmetic industry.

In the preparation of this standard, assistance has been obtained from cosmetic, Toiletry and Fragrance Association. CTFA Specification (formerly the Toilet Goods Association) Specification No. 9 'Beeswax', issued by the Board of Standards, Inc, New York. Reference has also been made to Indian Pharmacopoeia.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

**AMENDMENT NO. 1 MAY 2002
TO
IS 4028 : 1992 BEESWAX, BLEACHED FOR COSMETIC
INDUSTRY — SPECIFICATION
(Third Revision)**

[Page 1, Table 1 Sl No. (ii)] — Substitute the following for the existing:

(1)	(2)	(3)	(4)
(i)	Acid value	5-15	A-3

[Page 1, Table 1, Sl No. (iii), col 3] Substitute '87-104' for '85-95'.

[Page 1, Table 1, Sl No. (viii)] — Insert the following after Sl No. (viii):

(1)	(2)	(3)	(4)
ix)	Glycerin and other polyhydric alcohols	To pass the test ✓	A-10

(Page 3, clause A-4.2.4) — Substitute 'Indicator' for 'Indication'.

(Page 5, clause A-8.2, line 3) — Substitute '5.0 g' for '50 g'.

(Page 6, clause A-9.2) — Insert the following new clause after A-9.2:

A-10 TEST FOR GLYCERIN AND OTHER POLYHYDRIC ALCOHOLS

A-10.1 Reagents

A-10.1.1 Alcoholic Potassium Hydroxide Solution — approximately 0.5 N, prepared by dissolving potassium hydroxide in rectified spirit.

A-10.1.2 Sulphuric Acid — 1 M.

A-10.1.3 Sodium Periodate — 1.07 percent m/v.

A-10.1.4 Glycerin — 0.001 percent m/v.

A-10.1.5 Fuchsin Solution

Amend No. 1 to IS 4028 : 1992

A-10.2 Procedure

Take 0.2 g of the sample and add 10 ml of alcoholic potassium hydroxide solution, heat under a reflux condenser in a water bath for 30 minutes. Add 50 ml of 1 M sulphuric acid. Cool and filter, rinse the flask with 1 M sulphuric acid and combine the filtrate and washings in a 100-ml flask. Make up the volume with 1 M sulphuric acid. Label this solution as Solution A. Into two matched test tubes, introduce respectively, 1 ml of solution A and 1 ml of glycerin solution in 1 M sulphuric acid (Solution B). Add 0.5 ml of solution of sodium periodate to each tube, mix and allow to stand for 5 minutes. Add to each tube 1 ml of decolourized fuchsin solution and mix so that any precipitate disappears. Place the tubes in a beaker containing water at 40°C and observe for 10 to 15 minutes.

A-10.2.1 The material shall be taken to have passed the test if any bluish violet colour in the tube containing Solution A is not more intense than that in the tube containing Solution B.

(PCD 19)

Indian Standard

BEESWAX, BLEACHED FOR COSMETIC INDUSTRY — SPECIFICATION

(Third Revision)

1 SCOPE

This standard prescribes the requirements and the methods of sampling and test for beeswax, bleached, of Indian origin, for cosmetic industry.

2 REFERENCES

The following Indian standards are necessary adjuncts to this standard:

IS No.	Title
323 : 1959	Rectified spirit (<i>revised</i>)
548 (Part 1) : 1964	Methods of sampling and tests for oil and fats, Part 1 Sampling, physical and chemical test (<i>revised</i>)
1070 : 1992	Reagent grade water — Specification (<i>third revision</i>)
2316 : 1990	Methods of preparation standard solutions for colorimetric and volumetric analysis
3958 : 1984	Methods of sampling cosmetics (<i>first revision</i>)
4707 (Part 1) : 1988	Classification for cosmetic raw materials and adjuncts: Part 1 Dyes, colours and pigments (<i>first revision</i>)
4707 (Part 2) : 1992	Classification for cosmetic raw materials and adjuncts: Part 2 List of Raw Materials generally not recognized as safe for use in cosmetics (<i>first revision</i>)

The above-mentioned standards contain provisions which, through reference in this text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated above.

3 TERMINOLOGY

For the purpose of this standard, the definitions given in IS 548 (Part 1) : 1964 shall apply.

4 REQUIREMENTS

4.1 Description

The material shall be white to light yellow (as agreed between the buyer and the seller) in colour and shall have a faint and characteristic odour. The material shall be in a form as agreed to between the purchaser and the supplier. In the absence of such an agreement, it shall be in the form of slabs.

4.2 Beeswax is insoluble in water and sparingly soluble in cold alcohol, completely soluble in chloroform, in ether, in fixed and volatile oils; and partly soluble in cold benzene and in cold carbon tetrachloride but completely soluble in these liquids at 30°C.

4.3 The material shall also comply with the requirements given in Table 1 when tested according to methods prescribed in Annex A. Reference to relevant clauses of Annex A is given in col 4 of the table.

**Table 1 Requirements for Beeswax,
Bleached for Cosmetic Industry**

Sl No.	Characteristics	Requirement	Method of Test (Ref to Cl No. in Annex A)
(1)	(2)	(3)	(4)
i)	Melting point, °C	61.0 to 65.0	A-2
ii)	Acid value, <i>Max</i>	10	A-3
iii)	Saponification value	85 to 95	A-4
iv)	Fats, fatty acids, Japan Wax	To pass test	A-5
v)	Ceresin, paraffin and other waxes	To pass test	A-6
vi)	Iodine value, <i>Max</i>	10	A-7
vii)	Ash, percent by mass, <i>Max</i>	0.1	A-8
viii)	Total volatile matter at 105°C, percent by mass, <i>Max</i>	1.0	A-9

5 PACKING AND MARKING

5.1 Packing

Unless otherwise agreed to between the purchaser and the supplier, each slab shall be wrapped in grease-proof paper or a suitable plastic material, like polyethylene film. A number of such slabs shall be packed together in a suitable container.

5.2 Marking

Each container shall be suitably marked to give the following information:

a) Name of the material;

- b) Indication of the source of manufacture;
- c) Batch number in code or otherwise; and
- d) Net mass.

5.2.1 The container may also be marked with the Standard Mark.

6 SAMPLING

The method of preparing representative test samples of the material and the criteria for conformity shall be as prescribed in IS 3958 : 1984.

ANNEX A

(Clause 4.3)

ANALYSIS OF BEESWAX, BLEACHED, FOR COSMETIC INDUSTRY

A-1 QUALITY OF REAGENTS

A-1.1 Unless specified otherwise, pure chemicals and distilled water (see IS 1070 : 1992) shall be used in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

A-2 DETERMINATION OF MELTING POINT

A-2.1 Apparatus

A-2.1.1 Thermometer of a suitable type, with an accuracy of 0.1°C and graduated at every 0.1°C.

A-2.1.2 *Test Tube* — with a centrally bored cork to take the thermometer. The cork shall have a slit so as to permit circulation of air.

A-2.1.3 *Water Bath* — of suitable type, with the thermometer.

A-2.2 Procedure

Melt the material by warming it in a water-bath at a temperature just sufficient to melt it. Dip the thermometer and withdraw, so as to get the bulb thinly coated with wax. Let it stand for 24 hours. Insert the thermometer into the test-tube through the bored cork and then place the test tube in the water bath. Raise the temperature gradually, at the rate of 1°C in 3 minutes. Note the temperature accurately

to 0.1°C, at which a transparent drop forms on the end of the thermometer bulb. Record this temperature as the melting point of the material.

A-3 DETERMINATION OF ACID VALUE

A-3.0 Outline of the Method

The acid value is determined by titrating the material in benzene-alcohol medium with potassium hydroxide solution.

A-3.1 Reagents

A-3.1.1 *Benzene* — neutral to phenolphthalein indicator.

A-3.1.2 *Rectified Spirit* — neutral to phenolphthalein indicator (see IS 323 : 1959).

A-3.1.3 *Standard Potassium Hydroxide Solution* — 0.5 N.

A-3.1.4 Phenolphthalein Indicator Solution

Dissolve 0.1 g of phenolphthalein in 60 ml of rectified spirit and dilute with water to 100 ml.

A-3.2 Procedure

Mix the material thoroughly, making it entirely liquid before weighing. Weigh accurately about 5 g of the material in a 250-ml conical flask. Add 75 ml of a mixture of two parts of benzene and one part of rectified spirit. Heat

under reflux until the sample dissolves. Allow it to cool to room temperature and titrate with standard potassium hydroxide solution using phenolphthalein as indicator.

A-3.3 Calculation

$$\text{Acid value} = \frac{56.1 V N}{M}$$

where

V = volume in ml of standard potassium hydroxide solution used;

N = normality of standard potassium hydroxide solution; and

M = mass in g of the material taken for the test.

A-4 DETERMINATION OF SAPONIFICATION VALUE

A-4.0 Outline of the Method

The material is saponified by refluxing with a known excess of alcoholic potassium hydroxide solution. The alkali consumed for saponification is determined by titrating the excess alkali with standard acid.

A-4.1 Apparatus

A-4.1.1 Conical Flasks — 250 to 300 ml capacity, made of alkali-resistant glass.

A-4.1.2 Reflux Air Condenser — at least 65 cm long.

A-4.2 Reagents

A-4.2.1 Methyl Ethyl Ketone — This shall be stored in a dark place.

A-4.2.2 Rectified Spirit — same as in A-3.1.2.

A-4.2.3 Alcoholic Potassium Hydroxide Solution

Dissolve 30 g of potassium hydroxide in rectified spirit and make up to 1 litre. Allow to settle overnight in a dark place, decant the clear liquid and keep in a bottle closed tight with cork or rubber stopper.

A-4.2.4 Phenolphthalein Indication Solution — same as in A-3.1.4.

A-4.2.5 Standard Hydrochloric Acid — 0.5 N.

A-4.3 Procedure

Weigh accurately about 2.0 g of the material in a tared conical flask. Add 25 ml of methyl ethyl ketone, followed by 25 ml of alcoholic potassium hydroxide solution. Add a few pieces of pumice stone and connect the reflux air condenser to the flask. Heat the flask on a water bath or electric hot plate for about 2

hours. Boil steadily but gently. After the flask and condenser have cooled somewhat, wash down the inside of the condenser with about 10 ml of rectified spirit. Add about 1 ml of phenolphthalein indicator solution and titrate with standard hydrochloric acid. Carry out a blank determination at the same time.

A-4.4 Calculation

$$\text{Saponification value} = \frac{56.1 (B - S) N}{M}$$

where

B = volume in ml of standard hydrochloric acid required for the blank;

S = volume in ml of standard hydrochloric acid required for the material;

N = normality of standard hydrochloric acid; and

M = mass in g of the material taken for the test.

A-5 TEST FOR FATS, FATTY ACIDS, JAPAN WAX AND ROSIN

A-5.1 Reagents

A-5.1.1 Sodium hydroxide Solution — 10 percent (m/v).

A-5.1.2 Dilute Hydrochloric Acid — approximately 4 N.

A-5.2 Procedure

Boil 5.0 g of the material for about 10 minutes with 80 ml of sodium hydroxide solution. Replace the water lost by evaporation. Cool and filter the solution through glass wool or asbestos pad. Make the filtrate acidic with dilute hydrochloric acid.

A-5.2.1 The material shall be taken to have passed the test even if the solution may show some opalescence but no precipitate is produced.

A-6 TEST FOR CERESIN, PARAFFIN AND OTHER WAXES

A-6.1 Reagents

A-6.1.1 Alcoholic Potassium Hydroxide Solution — approximately 0.5 N, prepared by dissolving potassium hydroxide in rectified spirit.

A-6.1.2 Rectified Spirit Aldehyde Free — see IS 323 : 1959.

Procedure for preparing aldehyde free spirit in laboratory.

To 125 ml alcohol contained in 1 000 ml flask, add 375 ml of dinitrophenylhydrazine solution, heat on a water bath under a reflux condenser for twenty-four hours, remove the alcohol by distillation, dilute to 1 000 ml with a 2 percent v/v solution of sulphuric acid, and set aside for 24 hours, no crystals shall produce.

A-6.2 Procedure

Place 3.00 g of the material in a round-bottom, 100 ml boiling flask fitted with a ground glass joint. Add 30 ml of a solution prepared by dissolving 40 g of potassium hydroxide in about 900 ml of aldehyde-free alcohol maintained at a temperature not exceeding 15°C and then when solution is complete, warming to room temperature and adding aldehyde-free alcohol to make 1 000 ml. Reflux the mixture gently for 2 hours. At the end of this period, open the flask, insert a thermometer into the solution, and place the flask in a container of water at a temperature of 80°C. Rotate the flask in the bath so that solution gets cool.

A-6.2.1 The material shall be taken to have passed the test if the solution shows no cloudiness or globule formation before the temperature reaches 65°C.

A-7 DETERMINATION OF IODINE VALUE

A-7.0 Outline of the Method

The material is treated with a known excess of iodine monochloride solution in glacial acetic acid. The excess of iodine monochloride is determined iodometrically.

A-7.1 Apparatus

A-7.1.1 Thermometer

An engraved stem thermometer, calibrated between 10°C and 65°C in 0.1°C intervals and with the 0°C point marked on the stem, is recommended. The thermometer shall have an auxiliary reservoir at the upper end, length of about 370 mm and diameter of about 6 mm.

A-7.2 Reagents

A-7.2.1 Carbon Tetrachloride or Chloroform

A-7.2.2 Acetic Acid

Glacial, 99 percent, having a melting point of 14.8°C and free from reducing impurities. Determine the melting point of the acetic acid and test it for reducing impurities as follows:

- a) Melting point determination — Take a 15-cm long test tube and fill it to about two-thirds with the acetic acid. Insert into the acid a thermometer satisfying

the requirements specified in A-7.1.1 through a cork stopper fitting the test-tube. The amount of acid should be at least double the quantity required to cover the bulb of the thermometer when the bottom of the latter is 12 mm from the bottom of the test tube. Suspend this tube within a larger test-tube through a cork. Cool the acid by immersing the assembly in ice water until the temperature is 10°C, then withdraw the assembly from the ice water and stir the acid rather vigorously for a few moments, thus causing the super-cooled liquid to crystallize partially and give a mixture of liquid and solid acid. Take thermometer readings every 15 seconds and consider that temperature at which the reading remains constant for at least two minutes as the true melting point.

- b) Test for reducing impurities (potassium permanganate test) — Dilute 2 ml of the acetic acid with 10 ml of water and add 0.1 ml of 0.1 N potassium permanganate solution and maintain at $27 \pm 2^\circ\text{C}$. The test shall be taken as having been satisfied if the pink colour is not discharged at the end of two hours.

A-7.2.3 Potassium Dichromate — finely ground.

A-7.2.4 Starch Solution

Titrate 5 g of starch and 0.01 g of mercuric iodide with 30 ml of cold water and slowly pour it with stirring into 1 litre of boiling water. Boil for 3 minutes. Allow the solution to cool and decant off the supernatant clear liquid.

A-7.2.5 Standard Sodium Thiosulphate Solution— 0.1 N (see IS 2316 : 1963).

A-7.2.6 Chlorine Gas — dry,

A-7.2.7 Iodine Trichloride

A-7.2.8 Wijs Iodine Monochloride Solution

Prepare this solution by one of the following two methods, and store in a glass-stoppered bottle in a cool place, protected from light and sealed with paraffin until taken for use:

- a) Dissolve 13 g of re-sublimed iodine in 1 litre of acetic acid, using gentle heat if necessary, and determine the strength by titration with standard sodium thiosulphate solution. Set aside 50 to 100 ml of the solution and introduce washed and dried chlorine gas into the remainder until the characteristic colour change occurs and the halogen content is nearly doubled as ascertained again by titration.

If the halogen content has been more than doubled, reduce it by adding the requisite quantity of the iodine acetic acid solution. A slight excess of iodine does not harm, but avoid an excess of chlorine.

Example

If the titration of 20 ml of original iodine-acetic acid solution requires 22 ml of standard sodium thiosulphate solution, then 20 ml of the finished Wij's solution should require between 43 and 44 ml (and not more than 44 ml) of the same sodium thiosulphate solution.

- b) As an alternative method for preparing Wij's solution, dissolve 8 g of iodine trichloride in approximately 450 ml of acetic acid. Dissolve separately 9 g of iodine in 450 ml of acetic acid using heat if necessary. Add gradually the iodine solution to the iodine trichloride solution until the colour has changed to reddish brown. Add 50 ml more of iodine solution and dilute the mixture with acetic acid till 10 ml of the mixture are equivalent to 20 ml of standard sodium thiosulphate solution when the halogen content is estimated by titration in the presence of an excess of potassium iodide and water. Heat the solution to 100°C for 20 minutes and cool. Prevent access of water vapour in preparing the solution.

NOTE — The method prescribed in (a) is preferable to that prescribed in (b) because of the instability of iodine trichloride.

A-7.3 Procedure

Melt the material and filter through a filter paper to remove any impurities and the last traces of moisture. Make sure that the glass apparatus used is absolutely clean and dry. Weigh accurately, by difference, an appropriate quantity of the material as indicated in Table 2, into a clean, dry 500-ml glass stoppered bottle to which 25 ml of carbon tetrachloride or chloroform have been added, and agitate to dissolve the content. Add 25 ml of Wij's solution. (The quantity of Wij's solution added is 50 to 60 percent more than the quantity required). Replace the glass stopper after wetting with potassium iodide solution, swirl for intimate mixing, and allow to stand in the dark for 45 minutes. Carry out a blank test simultaneously under similar experimental conditions. After standing, add 15 ml of potassium iodide solution and 100 ml of water, and titrate the liberated iodine with standard

sodium thiosulphate solution, swirling the content of the bottle continuously to avoid any local excess, until the colour of the solution is straw yellow. Add 0.5 ml of starch solution and continue the titration until the blue colour disappears.

A-7.4 Calculation

$$\text{Iodine value} = \frac{12.69 (B - S) N}{W}$$

where

B = volume in ml of standard sodium thiosulphate solution required for the blank;

S = volume in ml of standard sodium thiosulphate solution required for the material;

N = normality of standard sodium thiosulphate solution; and

W = weight in g of the material taken for the test.

Table 2 Weight of the Material for Iodine Value Determination

(Clause 7.3)

Iodine Value Expected	Weighing		Weigh Accuracy in g
	Max	Min	
(1)	(2)	(3)	(4)
Less than 3	10.495	9.515	±0.001
3 to 5	6.346 0	5.077 0	±0.000 5
5 to 10	3.173 0	2.538 4	±0.000 2
10 to 50	0.661 2	0.528 8	±0.000 2

A-8 DETERMINATION OF ASH

A-8.1 Apparatus

A-8.1.1 Platinum Dish — having a capacity of 100 ml.

A-8.2 Procedure

Heat the platinum dish to redness, cool to room temperature in a desiccator and weigh. Take about 50 g of the material in a watch glass and weigh accurately. Transfer about three quarters of this quantity to the platinum dish and heat on a Bunsen burner so that the material burns gently at the surface. When about half of the material is burnt away, stop heating, cool and add the remainder of the material. Weigh the watch glass again and find, by difference, the exact mass of sample transferred to the platinum dish. Heat again as before till the material is completely charred. Incinerate

in a muffle furnace at 55°C to 650°C for 1 hour. Cool to room temperature in a desiccator and weigh. Repeat incineration, cooling and weighing until the difference between two successive weighings is less than one milligram.

A-8.3 Calculation

$$\text{Ash, percent by mass} = \frac{100 M_2}{M_1}$$

where

M_2 = mass in g of the ash; and

M_1 = mass in g of the material taken for the test.

A-9 DETERMINATION OF TOTAL VOLATILE MATTER AT 105°C

A-9.1 Procedure

Weigh accurately about 10 g of the material

in a suitable dish, previously dried and weighed, and place it in an oven maintained at $105 \pm 2^\circ\text{C}$ for 6 hours. Cool the dish in a desiccator and weigh with the lid on. Heat the dish again in the oven for 30 minutes. Repeat the process until the loss in mass between two successive weighings is less than one milligram. Record the constant mass obtained.

A-9.2 Calculation

$$\text{Total volatile matter at } 105^\circ\text{C, percent by mass} = \frac{100 (M_1 - M_2)}{M_1 - M_3}$$

where

M_1 = mass in g of the dish with the material before heating;

M_2 = mass in g of the dish after heating; and

M_3 = mass in g of the empty dish.

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